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**Joining of Advanced and Specialty Materials IX**

**SESSION TOPIC: Joining of ceramics (including brazing)**

**PRESENTATION TYPE: Contributed (Oral)**

**TITLE: Fabrication and Characterization of Diffusion Bonds for Silicon Carbide**

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**ABSTRACT:** Diffusion bonds of silicon carbide (SiC) were fabricated using several different types of titanium (Ti) based interlayers between the SiC substrates. The interlayers were an alloyed Ti foil, a pure Ti foil, and a physically vapor deposited (PVD) Ti coating. Microscopy was conducted to evaluate the cross-sections of the resulting bonds. Microprobe analysis identified reaction formed phases in the diffusion bonded region. Uniform and well adhered bonds were formed between the SiC substrates. In the case where the alloyed Ti foil or a thick Ti coating (i.e. 20 micron) was used as the interlayer, microcracks and several phases were present in the diffusion bonds. When a thinner interlayer was used (i.e. 10 micron PVD Ti), no microcracks were observed and only two reaction formed phases were present. The two phases were preferred and fully reacted phases that did not introduce thermal stresses or microcracks during the cool-down stage after processing. Diffusion bonded samples were evaluated with the non-destructive evaluation (NDE) methods of pulsed thermography and immersion ultrasonic testing. Joined SiC substrates that were fully bonded and that had simulated bond flaws in the interlayer were also evaluated using immersion ultrasound. Pull testing was conducted on the bonds to determine the tensile strength. To demonstrate the joining approach for a complex multilayered component for a low NO<sub>x</sub> injector application, the diffusion bonding approach was used to join three 4" diameter SiC discs that contained complex fuel and air flow channels.



# Fabrication and Characterization of Diffusion Bonded Silicon Carbide

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VEHICLE TECHNOLOGY DIRECTORATE



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  - James Smith of QSS Group, Inc. at NASA GRC for conducting electron microprobe work.
  - Dr. Robert Okojie of NASA GRC for providing PVD Ti Coated CVD SiC.



## Outline

- **Application** – Micro-Electro-Mechanical Systems Lean Direct Injector (MEMS LDI) for Advanced Aircraft Gas Turbines
- **Joint Processing** - Diffusion Bonding With a Titanium Interlayer
- **Characterization** – SEM, Microprobe, NDE, Pull Test
- **Near Term Plans** – Subcomponent Fabrication
- **Summary and Conclusions**



# Joining Motivation



## Improve Ceramic to Ceramic and Ceramic to Metal Joining Methods

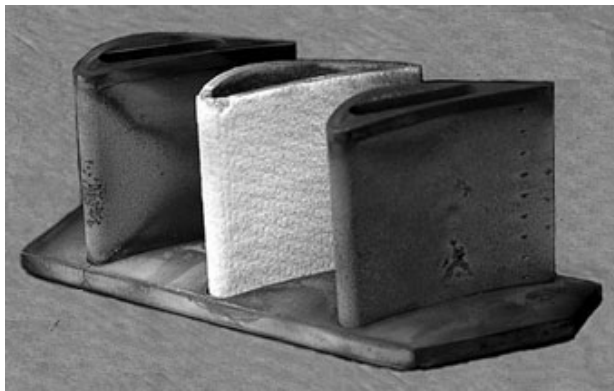
- allow for the fabrication of complex shapes and structures.
- allow for easier integration of ceramics components into engine systems.

Ceramics can be either monolithics or fiber reinforced composites.

## Examples of Component Applications Benefiting from Joining Technology

– components for aeronautic and ground based engine applications (i.e. ceramic turbine vanes, blades, injectors, rotors, combustor liners, valves, and heat shields) and fusion reactor components, optical space components, and bilayer armor.

**Benefits** – simplified component fabrication, enabling technology, increased operating temperatures, improved efficiency, reduced cooling requirements, reduced weight, higher creep resistance, and higher strength than current technologies.

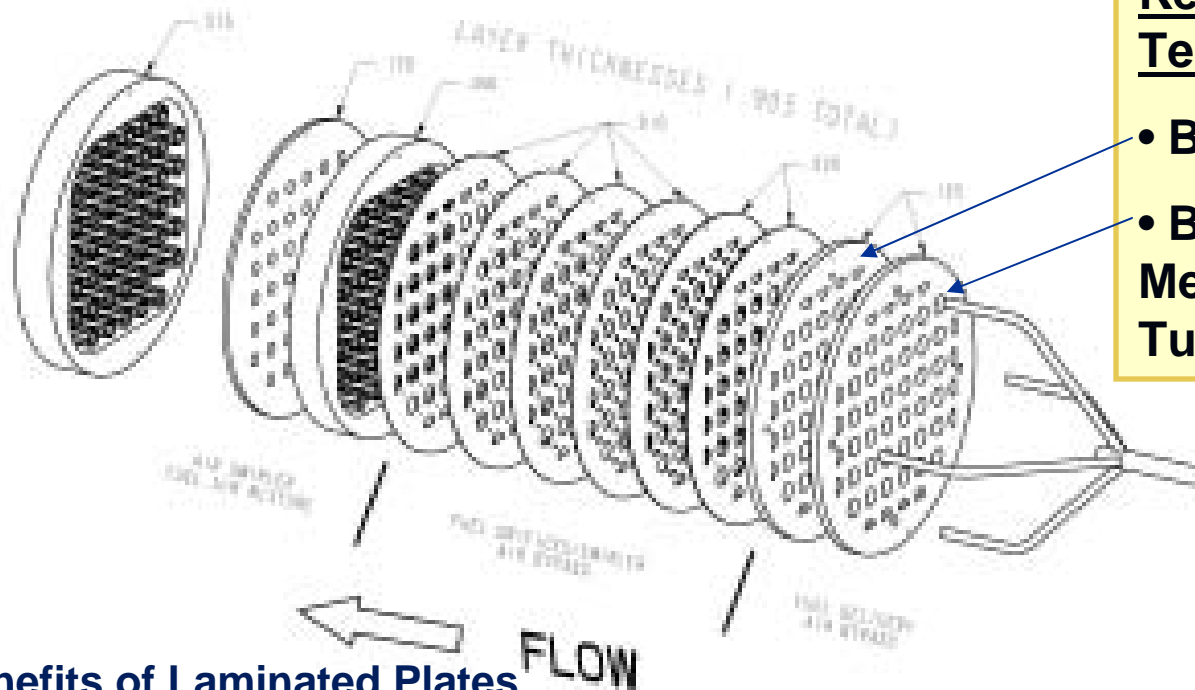




# Application - Lean Direct Injector Fabricated by Diffusion Bonding of SiC Laminates



SiC laminates can be used to create intricate and interlaced passages to speed up fuel-air mixing to allow lean-burning, ultra-low emissions



## Key Enabling Technologies:

- Bonding of SiC to SiC
- Brazing of SiC to Metallic (Kovar) Fuel Tubes

## Benefits of Laminated Plates

- Passages of any shape can be created to allow for multiple fuel circuits
- Provides thermal protection of the fuel to prevent choking
- Low cost fabrication of modules with complicated internal geometries through chemical etching



# Current Approach of Joining SiC With a Ti Layer



## Advantages of Diffusion Bonding Using a Ti Layer

- Uniform Ti layers can be applied
- Ti can be applied by different methods (foil, PVD and other coating approaches)
- High strength and leak-free bonds
- Good high temperature stability

**The objective is to develop joining technology that has the following capabilities necessary for the injector application:**

- Joining of relatively large geometries (i.e. 4" diameter discs)
- Leak-free at an internal pressure of 200 psi (1.38 MPa)
- Stability and strength retention at 800°F (427°C)



# SiC-Ti-SiC Diffusion Bond Processing Matrix



## SiC and Ti Material Combinations:

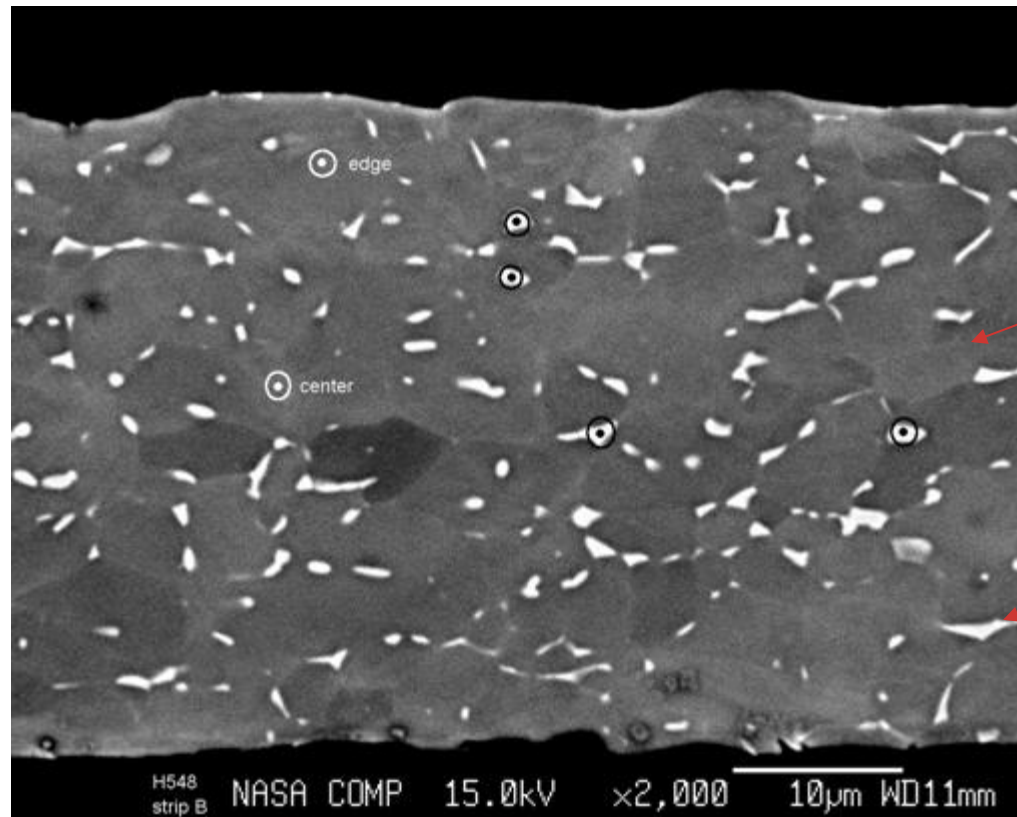
1. 1.75" diameter  $\alpha$ -SiC (CRYSTAR from Saint-Gobain) discs joined with a 38 micron alloyed Ti foil
2. 1.75" diameter CVD SiC (TREX Enterprises) discs joined with a 38 micron alloyed Ti foil
3. 1" x 2" CVD SiC (Rohm & Hass) coupons joined with ~10 micron PVD Ti coating on one of the surfaces
4. 1" x 2" CVD SiC (Rohm & Hass) coupons joined with a 38 micron alloyed Ti foil
5. 1" x 2" CVD SiC (Rohm & Hass) coupons joined with ~10 micron PVD Ti coating on both of the surfaces (20 micron total PVD Ti)

Condition	Temp. (°C)	Pressure* (MPa)	Time (hr)	Atmosphere	Cooling Rate (°C/min)	Analysis
A (materials 1, 2, and 3)	1250	24, 24, 31	2	vacuum	5	Microscopy & Microprobe
B (materials 1 and 3)	1300	24, 31	2	vacuum	2	Microscopy
C (materials 1 and 3)	1250	50	2	vacuum	2	Microscopy
D (materials 1, 4 and 5)	1250	24, 31	2	vacuum	2	Microscopy & Microprobe
*at minimum clamping pressure for the hot press						





## Alloyed Ti Foil



**Ti-6Al-4V (weight %)**

Grey phase – Alpha alloy

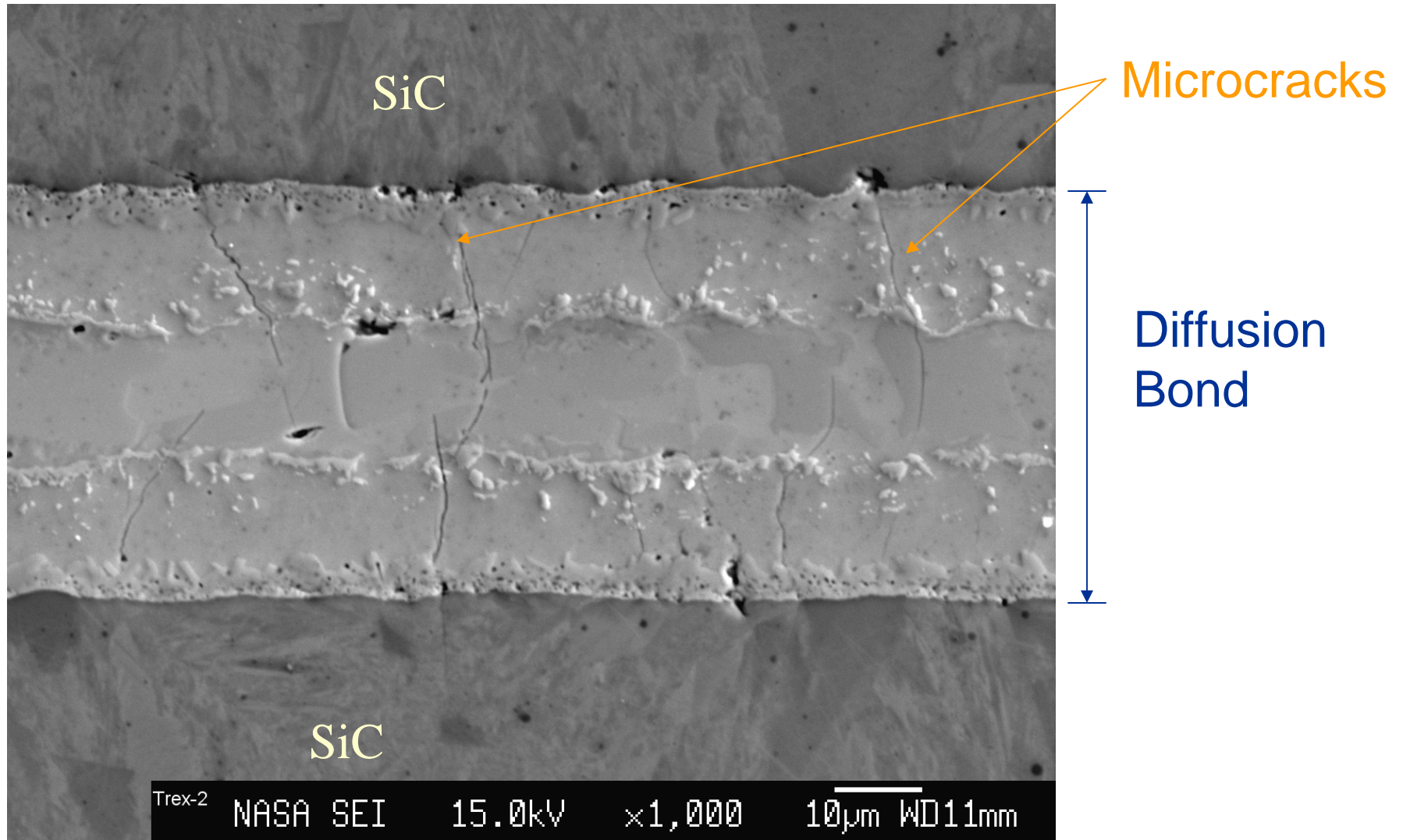
White phase – Beta alloy

Microprobe from the cross-section of alloyed Ti foil (averages taken from several points near the edge and at the center of the foil)

	Phase	Al	Fe	Ti	V	Total
Atomic Ratio	Grey Phase	10.196	0.042	86.774	2.988	100.000
Weight (%)	Grey Phase	5.999	0.051	90.632	3.318	100.000
Atomic Ratio	White Phase	4.841	1.850	76.507	16.803	100.000
Weight (%)	White Phase	2.748	2.172	77.084	17.997	100.000



## Secondary Electron Image of the Diffusion Bond - Alloyed Ti Foil and Trex CVD SiC

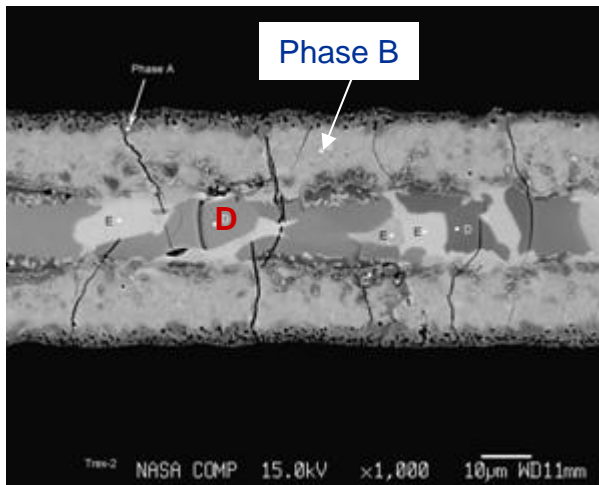




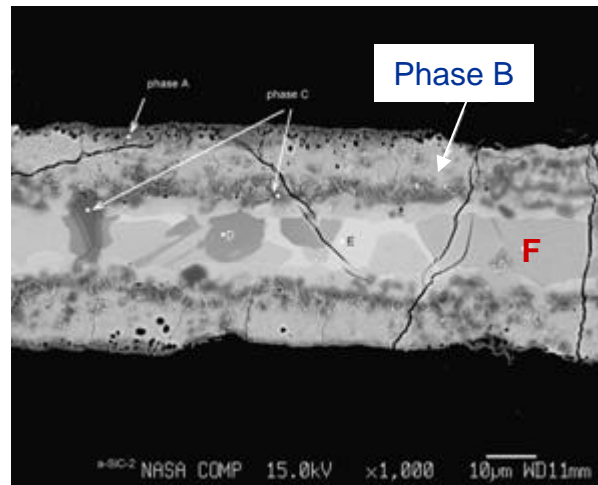
## Bonding with the Alloyed Ti Foil Between Different SiC Substrates



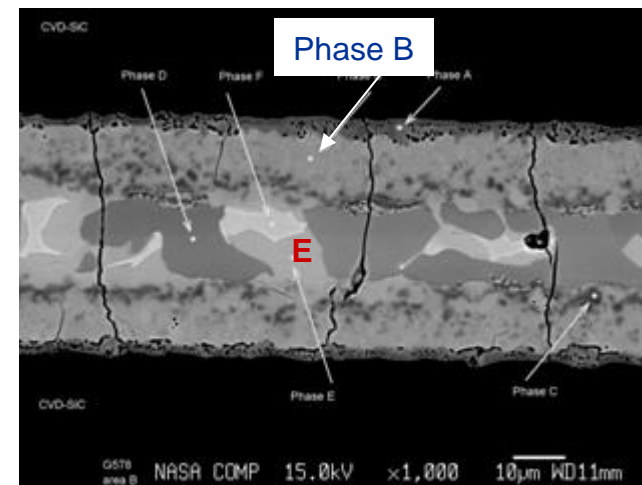
TREX CVD SiC



CRYSTAR  $\alpha$ -SiC



Rohm & Haas CVD SiC



Microcracks formed regardless of the substrate and variations in the processing: higher temperatures, higher pressures, slower cooling rate.

Microcracking may be due to thermal stresses during cooling down from processing:

- Phase B (same in all three micrographs) -  $\text{Ti}_5\text{Si}_3\text{C}_x$  ( $\text{Ti}_5\text{Si}_3$ ) is highly anisotropic in its thermal expansion where  $\text{CTE}(c)/\text{CTE}(a) = 2.72$  (Schneibel et al).
- Central core of diffusion bond has concentrated alpha and beta Ti alloy phases. The alpha phase has an anisotropic thermal expansion which is 20 % greater along the c-axis (Boyer, Welsch, and Colling). Also, the beta phase has a thermal expansion that is 6 x higher in the temperature range of 600-1000°C ( $5.8 \times 10^{-5}/^\circ\text{C}$ ) compared the thermal expansion below 600°C ( $9.2 \times 10^{-6}/^\circ\text{C}$ ) (Elmer et al).



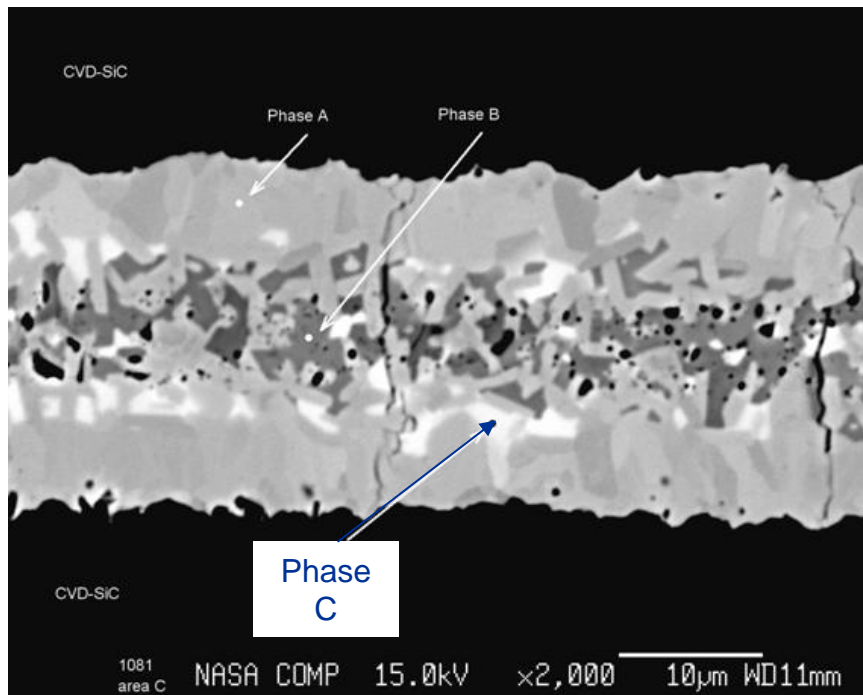
# Diffusion Bonds from Using PVD Ti as the Interlayer



## 20 Micron Ti Interlayer

Microcracking is still present due to the presence of  $\text{Ti}_5\text{Si}_3\text{C}_x$ .

Naka et al suggest that this is an intermediate phase.



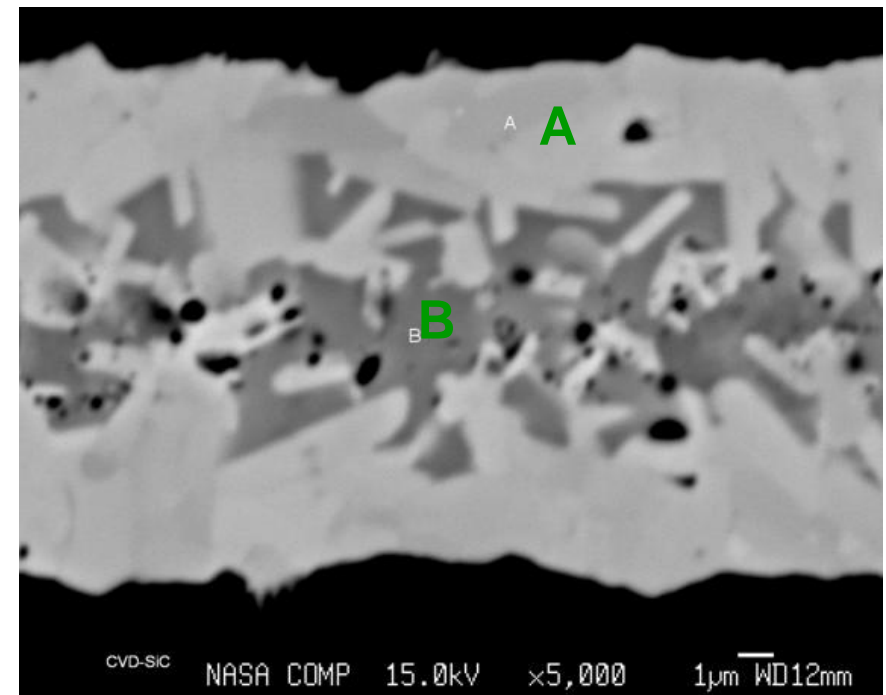
Phases in bond with the 20  $\mu$  Ti Interlayer – Atomic Ratios

Phase	Ti	Si	C
Phase A	56.426	17.792	25.757
Phase B	35.794	62.621	1.570
Phase C	58.767	33.891	7.140

## 10 Micron Ti Interlayer

No microcracking or phase of  $\text{Ti}_5\text{Si}_3\text{C}_x$  is present.

**Thin interlayers of pure Ti down-selected as the preferred interlayer.**

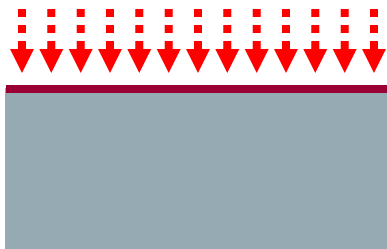
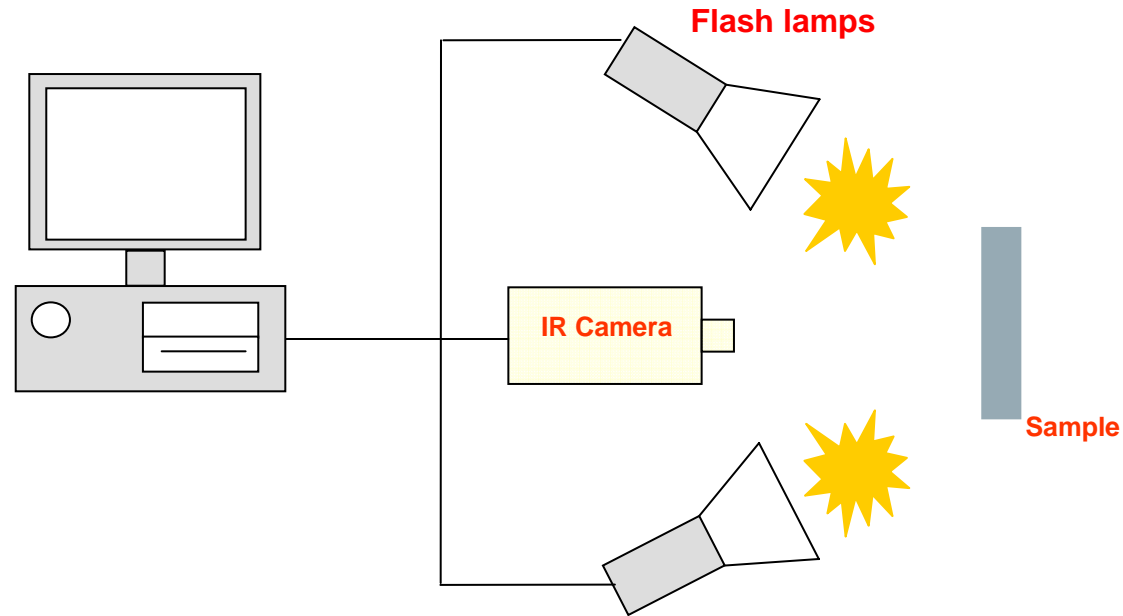


Phases in bond with the 10  $\mu$  Ti Interlayer – Atomic Ratios

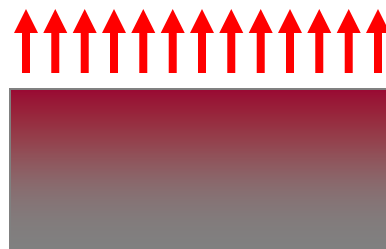
Phase	Ti	Si	C
SiC	0.011	54.096	45.890
Phase A	56.621	18.690	24.686
Phase B	35.752	61.217	3.028



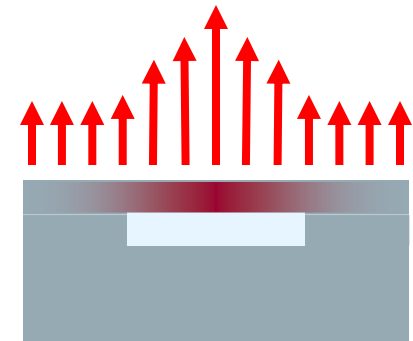
# Pulsed Thermography Overview



Light pulse uniformly heats sample surface

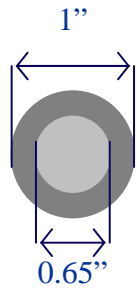


Thermal energy diffuses into the sample and is uniformly emitted from surface



Increase IR radiation at the surface due to the presence of a subsurface defect





# Pulsed Thermography Results of Bonded SiC Derivative Images

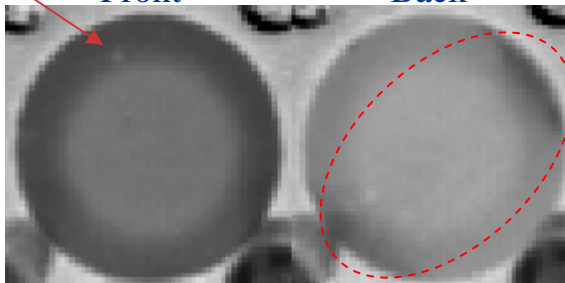


Visible from  
surface

## Unpolished

Front

Back



Run #113/31967 (On 106 Mini Hot Press)  
Thermal Derivative Image (0.007 s)



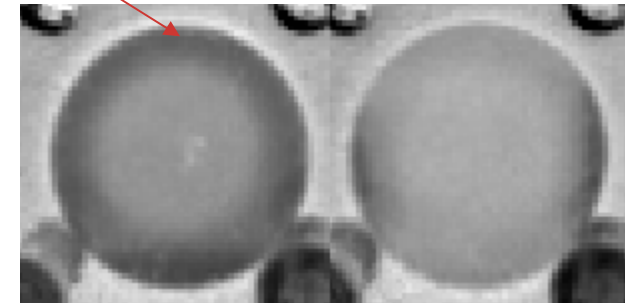
Optical Photos

Notch on front

## Polished

Front

Back



Run #114/31967  
Thermal Derivative Image (0.007 s)



Optical Photos

- Presence of bonding was detected in the pulsed thermography images from front and back of the SiC disks
- Results regarding bond quality are inconclusive due to surface conditions (i.e. reflections)

### Thermal Parameters:

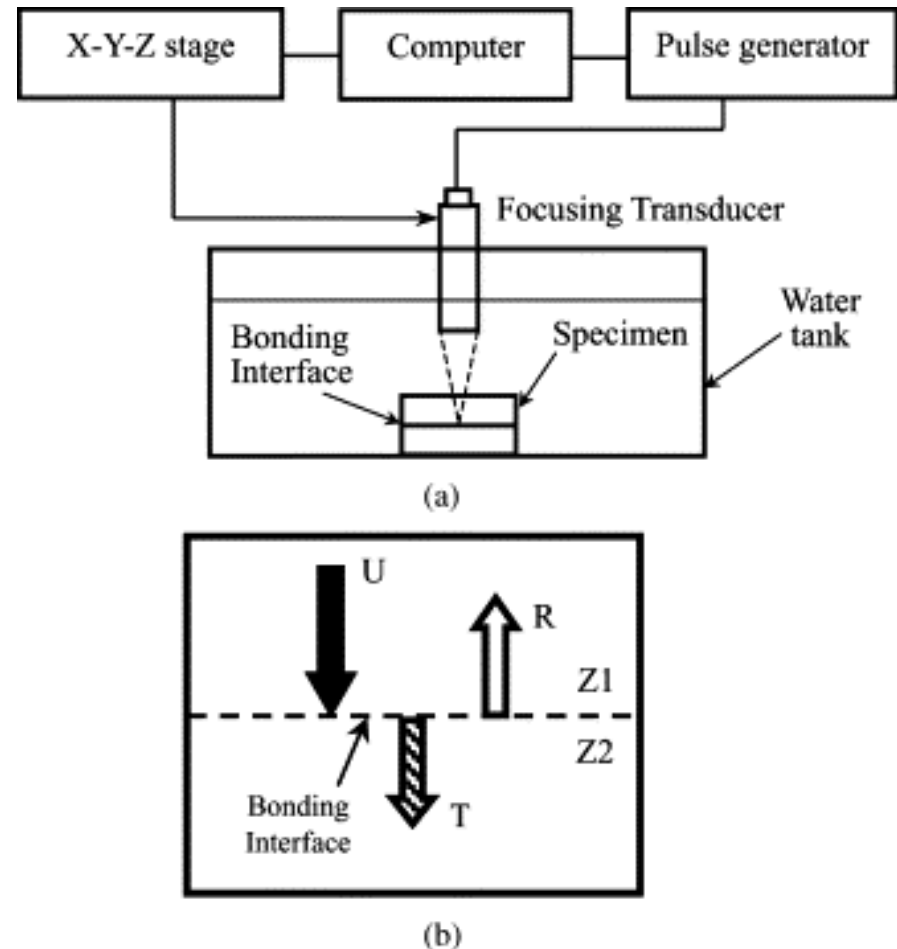
160x128 portion of focal plane array  
Capture rate: 409 Hz  
223 frames (0.5 s)



# Immersion Ultrasonic Testing Basics



- A high frequency ultrasonic pulse (U) enters specimen.
- Some of the signal is reflected back at the bond interface (R) while the remaining energy is transmitted (T) and eventually reflected at the back surface of the sample.
- The amount of energy reflected at the interface depends on the acoustic impedance (z) mismatch between the materials at the interface.
- Well bonded, similar materials will result in very little or no reflection at the interface while a disbond or air gap will cause more of the energy at the interface to reflect back.

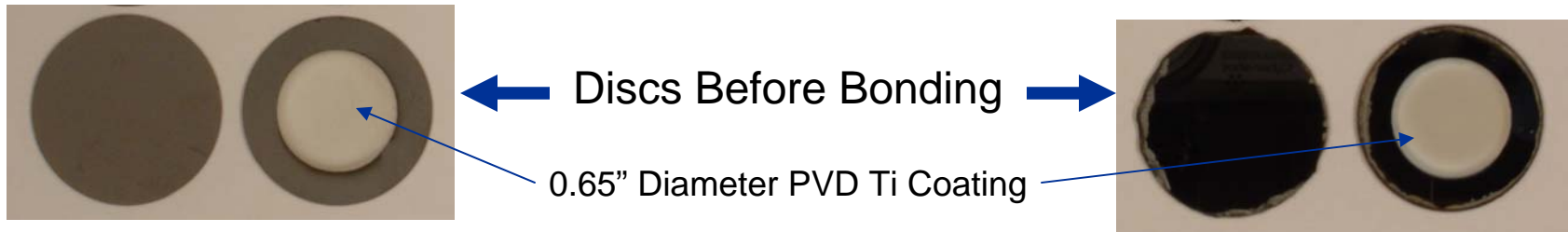




## Non-Destructive Evaluation (NDE) Method of Ultrasonic Immersion Shows Very Good Bonding of 1" Discs

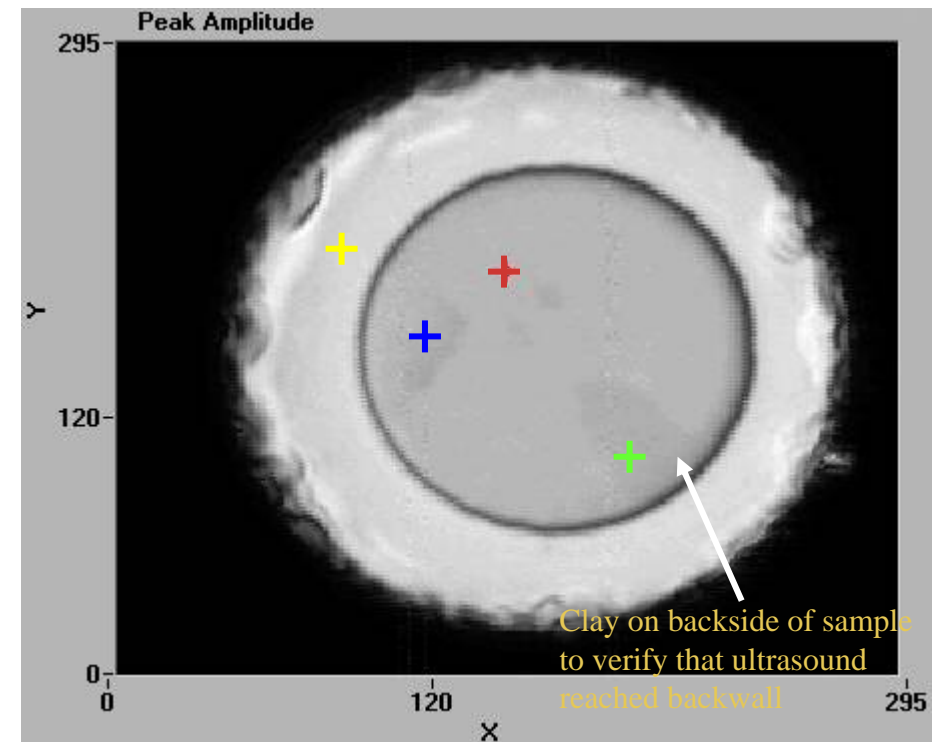
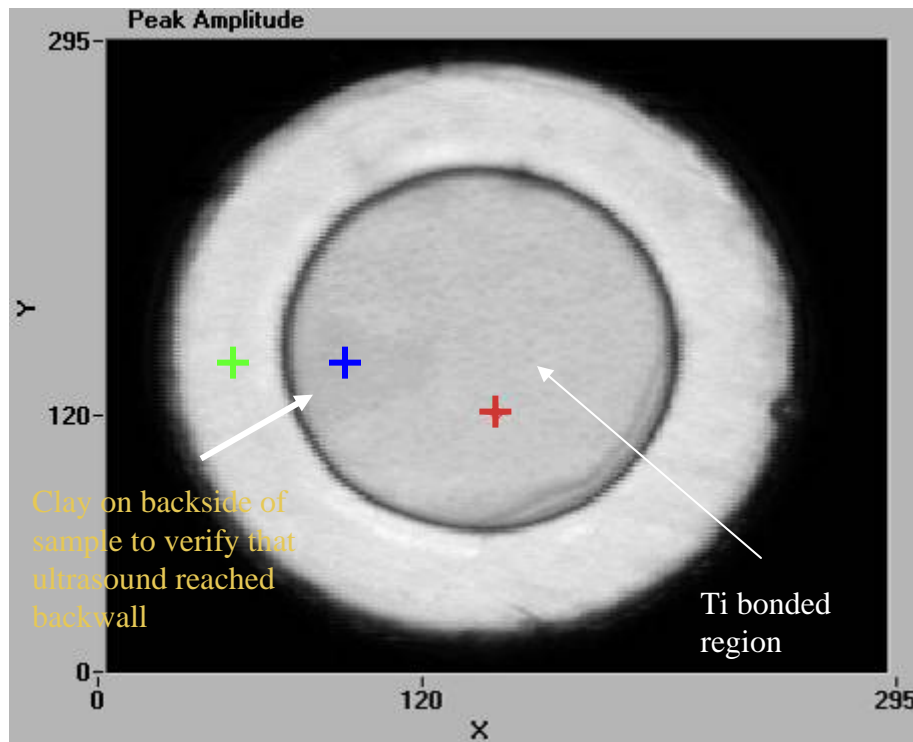
Less Polished SiC

Highly Polished SiC



Ultrasonic C-scan Image of Bonded Discs

Ultrasonic C-scan Image of Bonded Discs

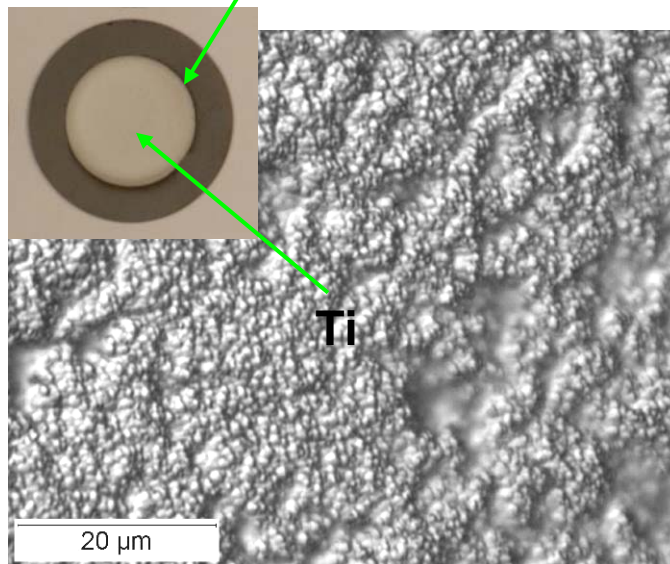
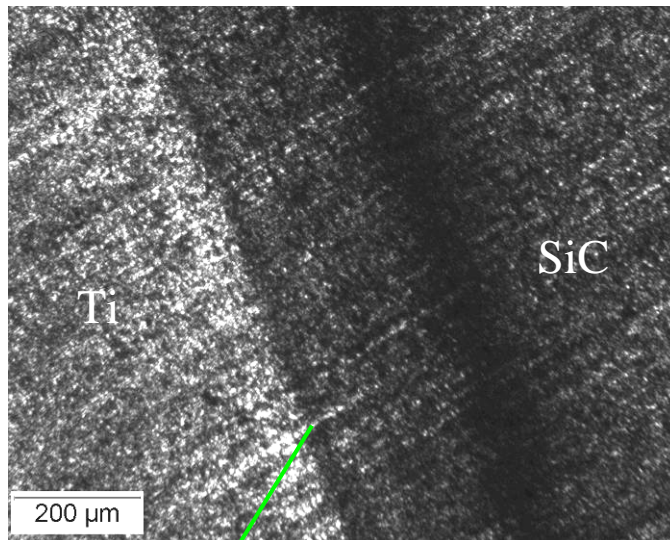




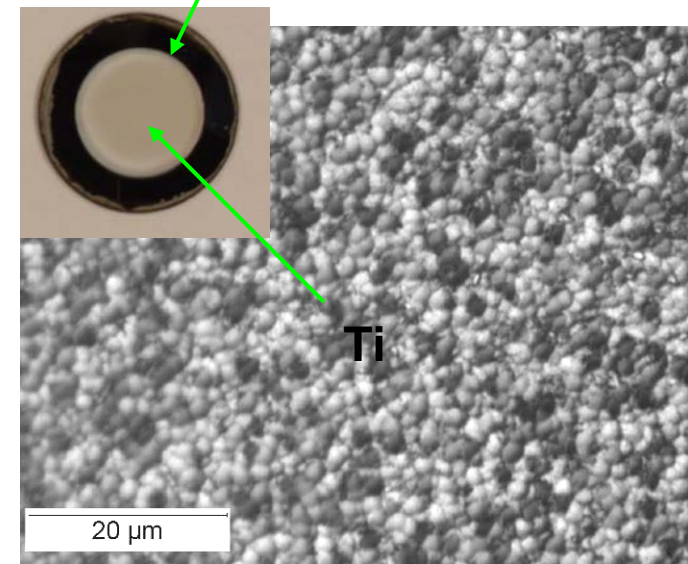
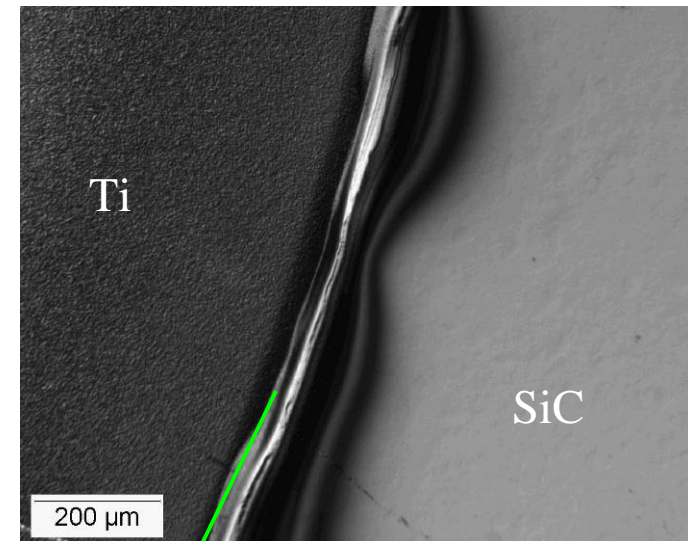


## Close-up View of PVD Ti Coatings on SiC

### On Less Polished SiC



### On Highly Polished SiC



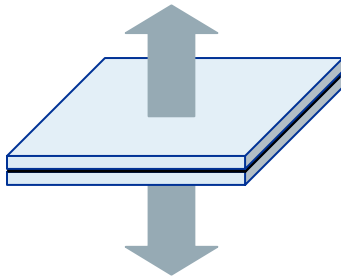
The coating is more dense on the highly polished SiC.



# The High Strength Bonds Greatly Exceed the Application Requirements



1" x 1" Bonded Substrates



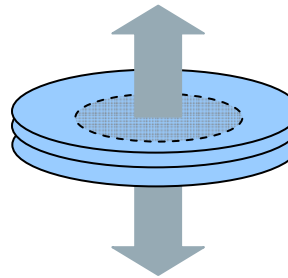
Pull test tensile strengths:

> 23.6 MPa (3.4 ksi)\*

> 28.4 MPa (4.1 ksi)\*

\* failure in the adhesive to the test fixture

1" Diameter Discs with a  
0.65" Diameter Bond Area



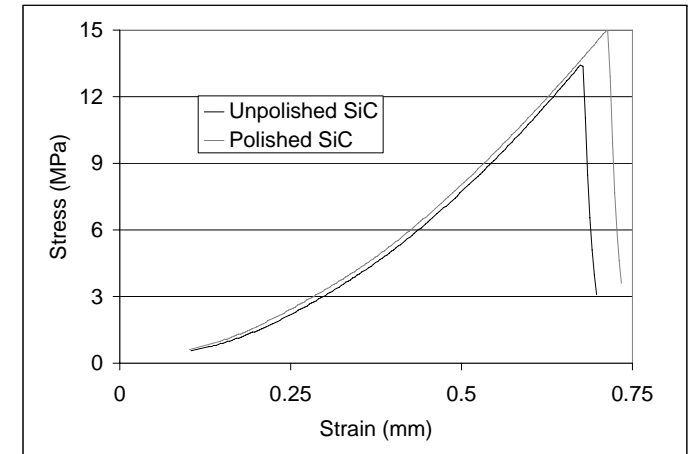
Pull test tensile strengths:

13.4 MPa (1.9 ksi)

15.0 MPa (2.2 ksi)

Slightly higher strength from the highly polished SiC suggest that a smoother surface contributes to stronger bonds or less flawed SiC.

Failures are primarily in the SiC substrate rather than in the bond area.



The injector application requires a strength of about 3.45-6.89 MPa (0.5 - 1.0 ksi).

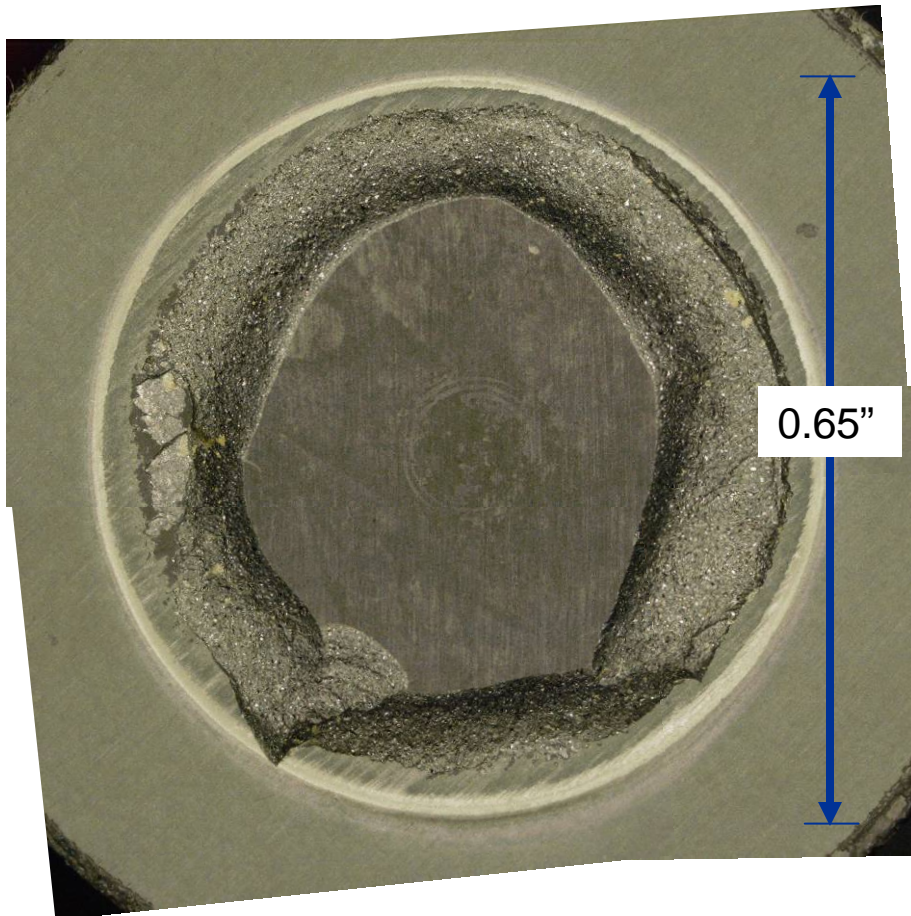




## Fracture Surfaces from the Pull Tests of the 1.0" Joined Discs with 0.65" Diameter Bond Region



Less Polished SiC



Failure was in the SiC substrate rather than the bond. The bond was pulled out intact from the failing SiC substrate.

Highly Polished SiC



Failure was primarily in the SiC as failure started in one substrate crossed through the bond region and continued in the other SiC substrate.



## Ultrasonic Immersion on Bonded Hexoloy SiC with Known/Simulated Flaws

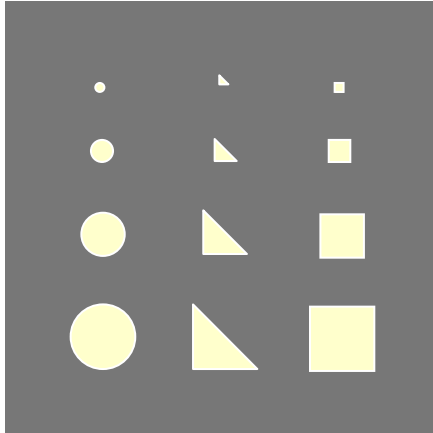
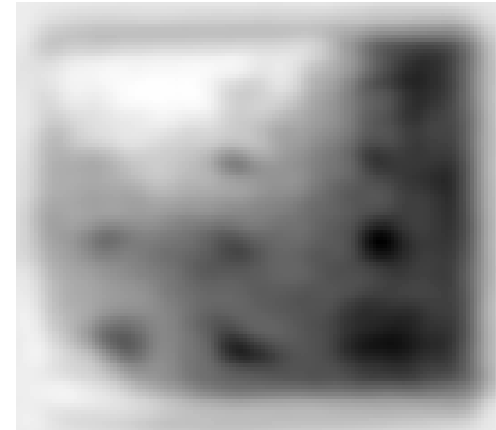


Illustration of Ti foil (grey) with cut patterns (white).

Two sets 1" x 1" SiC substrates were joined with a Ti interlayer that had different sized patterns cut out.



Ultrasonic C-scan @10MHz reveals areas of no bonding with a slightly increased reflection at the bond interface. Contrast is limited.

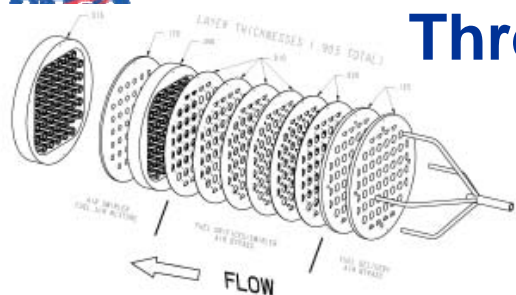
The poor NDE images may be due to:

- thicker SiC substrates - 0.25" compared to the 60 mill thick CVD SiC
- the lower density - 3.1 g/cm<sup>3</sup> vs. 3.21 g/cm<sup>3</sup> for the CVD SiC
- higher porosity 2-3% closed porosity vs. no porosity for the CVD SiC





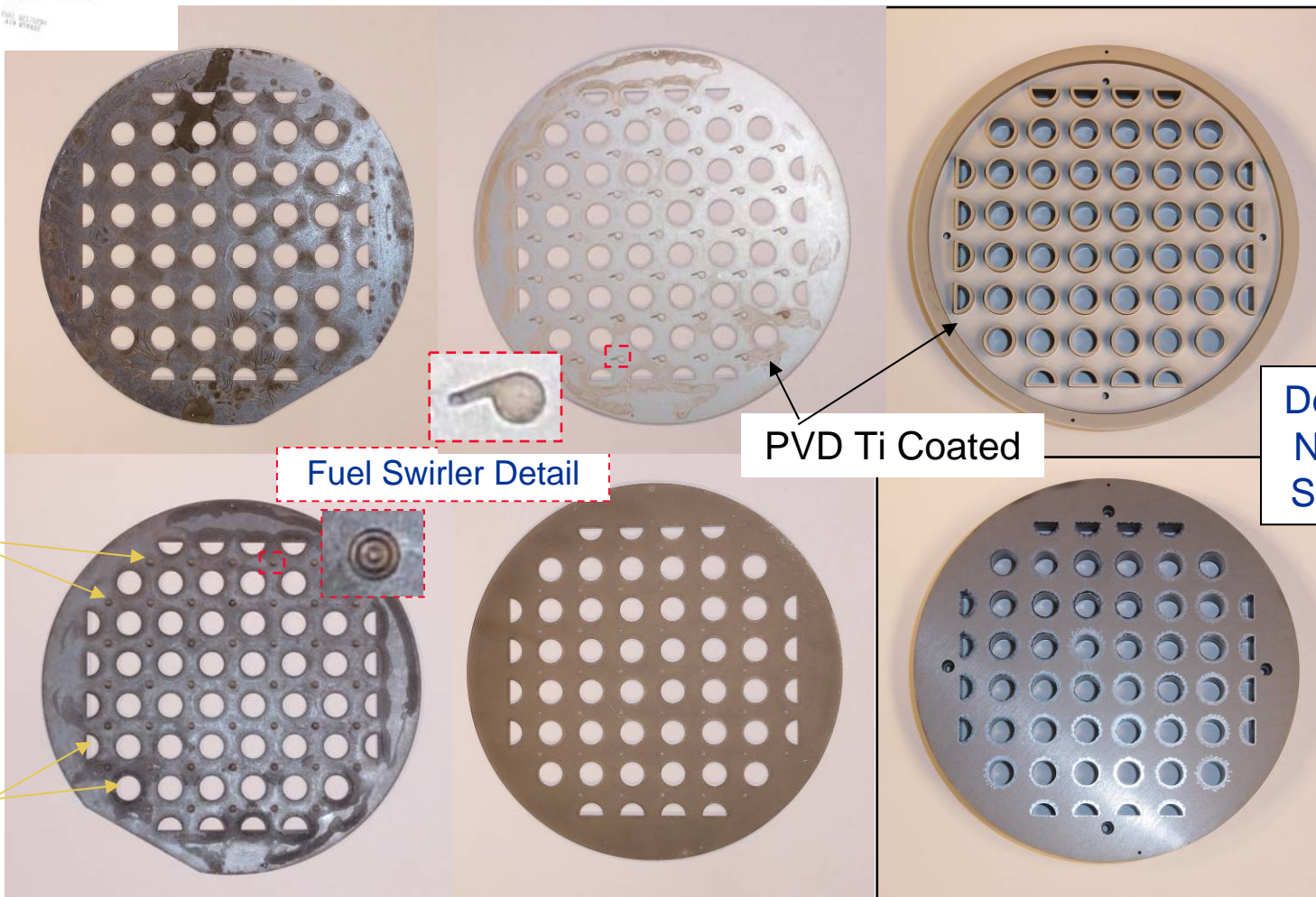
# Three Part 10 cm (4") Diameter SiC Injector



Stacking Sequence  
Top to Bottom



**Top  
Surfaces**



**Small Fuel  
Holes**

**Bottom  
Surfaces**

**Large Air  
Holes**

**Fuel Swirler Detail**

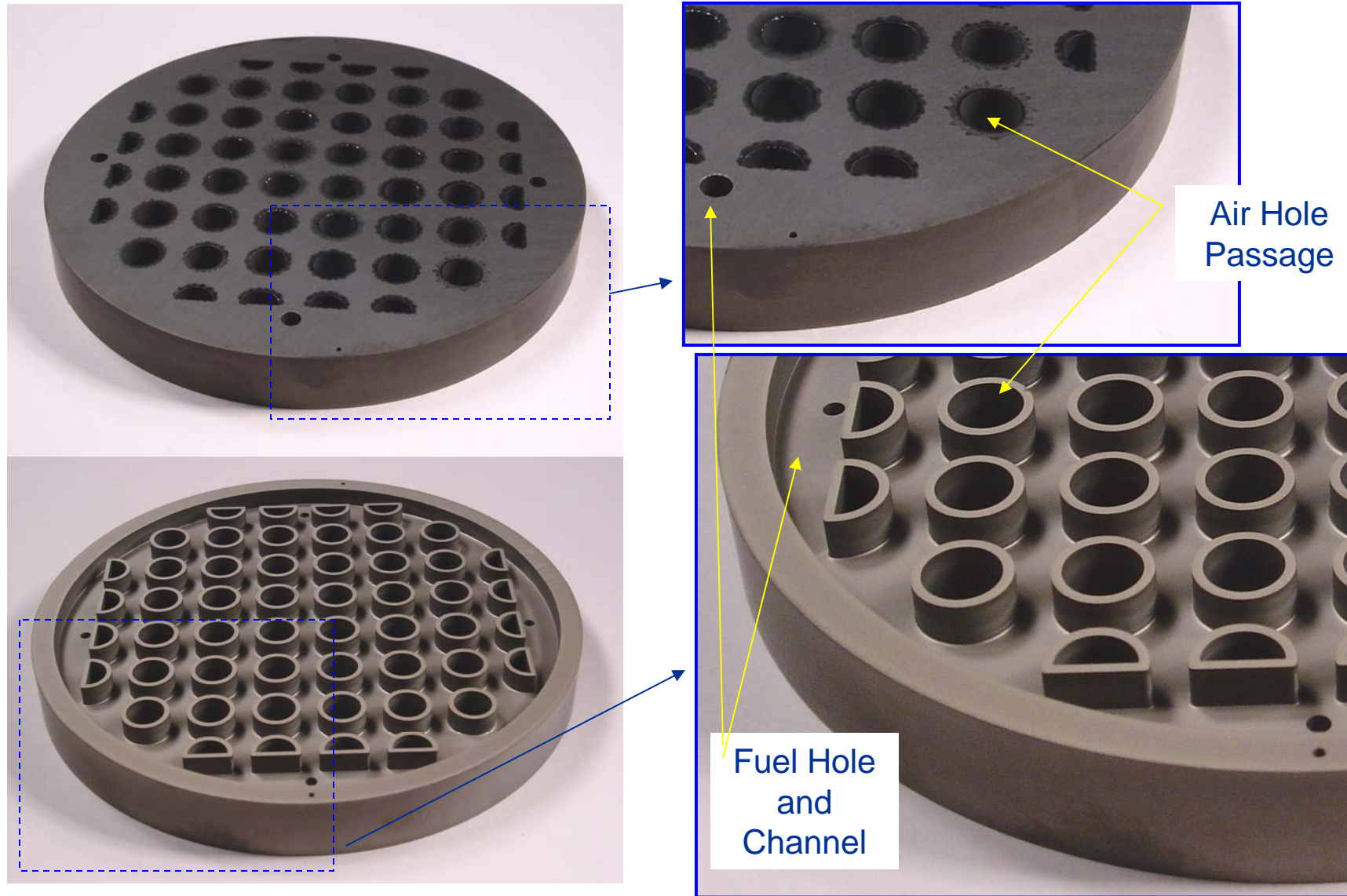
**PVD Ti Coated**

**Detail  
Next  
Slide**





## Detail of the Thickest Injector Substrate (~0.635 cm thick)





# Summary of Results and Future Studies

## Summary of Results:

- Diffusion bonds fabricated with the alloyed Ti foil as the interlayer formed five to seven reaction formed phases in the bond.
  - Microcracks due to the formation of thermally anisotropic phases.
- Diffusion bonds fabricated with the 10 micron PVD Ti coating gave better diffusion bonds than the alloyed Ti foil.
  - Fewer and preferred phases were formed which resulted in bonds without microcracks.
- NDE method of ultrasonic immersion shows good, uniform bonding.
- Strengths are well above the application requirements.

## Future Studies:

- Future efforts will further evaluate this bonding method to determine if it is fully capable of meeting the needs of the proposed injector application – uniform, leak-free bonds with stability and strength retention at temperatures up to 800°F.
- Develop and characterize Kovar to SiC joining.
- Apply joining techniques to component fabrication and support in-house component testing.